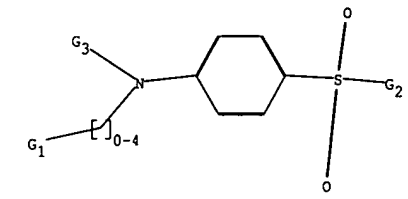
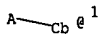
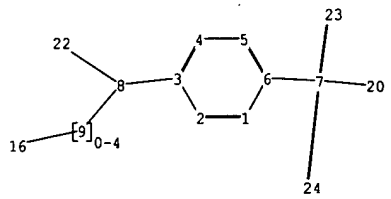
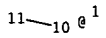
C:\Program Files\Stnexp\Queries\09844061.str

Structure search after final amend.
2/18/03









chain nodes :
7 8 9 10 11 12 16 20 22 23 24
ring nodes :
1 2 3 4 5 6
chain bonds :
3-8 6-7 7-20 7-23 7-24 8-9 8-22 9-16 10-11
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6
exact/norm bonds :
3-8 6-7 7-20 7-23 7-24 8-9 8-22 9-16 10-11
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6

G1:[*1],[*2]

G2:C,N,Cb

G3:C,Cy

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:CLASS 8:CLASS 9:CLASS 10:Atom
11:CLASS 12:Atom 16:CLASS 20:CLASS 22:CLASS 23:CLASS 24:CLASS

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:ssspta1611hxl

PASSWORD:
TERMINAL (ENTER 1, 2, 3, OR ?):2

* * * * * * * * * * Welcome to STN International * * * * * * * * * *

NEWS 1		Web Page URLs for STN Seminar Schedule - N. America
NEWS 2	Apr 08	"Ask CAS" for self-help around the clock
NEWS 3	Apr 09	BEILSTEIN: Reload and Implementation of a New Subject Area
NEWS 4	Apr 09	ZDB will be removed from STN
NEWS 5	Apr 19	US Patent Applications available in IFICDB, IFIPAT, and IFIUDB
NEWS 6	Apr 22	Records from IP.com available in CAPLUS, HCAPLUS, and ZCAPLUS
NEWS 7	Apr 22	BIOSIS Gene Names now available in TOXCENTER
NEWS 8	Apr 22	Federal Research in Progress (FEDRIP) now available
NEWS 9	Jun 03	New e-mail delivery for search results now available
NEWS 10	Jun 10	MEDLINE Reload
NEWS 11	Jun 10	PCTFULL has been reloaded
NEWS 12	Jul 02	FOREGE no longer contains STANDARDS file segment
NEWS 13	Jul 22	USAN to be reloaded July 28, 2002; saved answer sets no longer valid
NEWS 14	Jul 29	Enhanced polymer searching in REGISTRY
NEWS 15	Jul 30	NETFIRST to be removed from STN
NEWS 16	Aug 08	CANCERLIT reload
NEWS 17	Aug 08	PHARMAMarketLetter(PHARMAML) - new on STN
NEWS 18	Aug 08	NTIS has been reloaded and enhanced
NEWS 19	Aug 19	Aquatic Toxicity Information Retrieval (AQUIRE) now available on STN
NEWS 20	Aug 19	IFIPAT, IFICDB, and IFIUDB have been reloaded
NEWS 21	Aug 19	The MEDLINE file segment of TOXCENTER has been reloaded
NEWS 22	Aug 26	Sequence searching in REGISTRY enhanced
NEWS 23	Sep 03	JAPIO has been reloaded and enhanced
NEWS 24	Sep 16	Experimental properties added to the REGISTRY file
NEWS 25	Sep 16	CA Section Thesaurus available in CAPLUS and CA
NEWS 26	Oct 01	CASREACT Enriched with Reactions from 1907 to 1985
NEWS 27	Oct 21	EVENTLINE has been reloaded
NEWS 28	Oct 24	BEILSTEIN adds new search fields
NEWS 29	Oct 24	Nutraceuticals International (NUTRACEUT) now available on STN
NEWS 30	Oct 25	MEDLINE SDI run of October 8, 2002
NEWS 31	Nov 18	DKILIT has been renamed APOLLIT
NEWS 32	Nov 25	More calculated properties added to REGISTRY
NEWS 33	Dec 02	TIBKAT will be removed from STN
NEWS 34	Dec 04	CSA files on STN
NEWS 35	Dec 17	PCTFULL now covers WP/PCT Applications from 1978 to date
NEWS 36	Dec 17	TOXCENTER enhanced with additional content
NEWS 37	Dec 17	Adis Clinical Trials Insight now available on STN
NEWS 38	Dec 30	ISMEC no longer available
NEWS 39	Jan 13	Indexing added to some pre-1967 records in CA/CAPLUS
NEWS 40	Jan 21	NUTRACEUT offering one free connect hour in February 2003
NEWS 41	Jan 21	PHARMAML offering one free connect hour in February 2003

NEWS 42 Jan 29 Simultaneous left and right truncation added to COMPENDEX, ENERGY, INSPEC
NEWS 43 Feb 13 CANCERLIT is no longer being updated

NEWS EXPRESS January 6 CURRENT WINDOWS VERSION IS V6.01a, CURRENT MACINTOSH VERSION IS V6.0b(ENG) AND V6.0Jb(JP), AND CURRENT DISCOVER FILE IS DATED 01 OCTOBER 2002
NEWS HOURS STN Operating Hours Plus Help Desk Availability
NEWS INTER General Internet Information
NEWS LOGIN Welcome Banner and News Items
NEWS PHONE Direct Dial and Telecommunication Network Access to STN
NEWS WWW CAS World Wide Web Site (general information)

Enter NEWS followed by the item number or name to see news on that specific topic.

* * * * * * * * * * * * * * STN Columbus * * * * * * * * * * * * * * * *

FILE 'HOME' ENTERED AT 16:24:02 ON 18 FEB 2003

=> fil reg

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.21	0.21

FILE 'REGISTRY' ENTERED AT 16:24:11 ON 18 FEB 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 17 FEB 2003 HIGHEST RN 491570-72-0
DICTIONARY FILE UPDATES: 17 FEB 2003 HIGHEST RN 491570-72-0

TSCA INFORMATION NOW CURRENT THROUGH MAY 20, 2002

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details:
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

=>
Uploading 09844061.str

L1 STRUCTURE UPLOADED

```
=> d l1
L1 HAS NO ANSWERS
L1                STR
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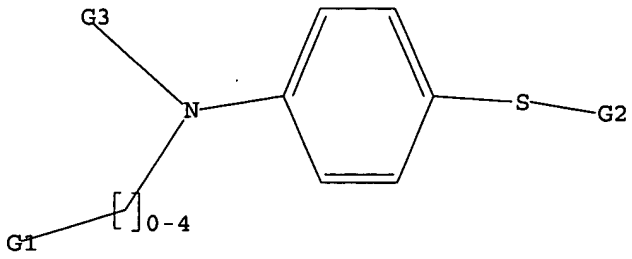
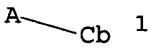
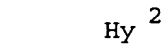






G1 [@1], [@2]

G2 C,N,Cb

G3 C,Cy

Structure attributes must be viewed using STN Express query preparation.

```
=> s l1
SAMPLE SEARCH INITIATED 16:24:29 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED -  14658 TO ITERATE

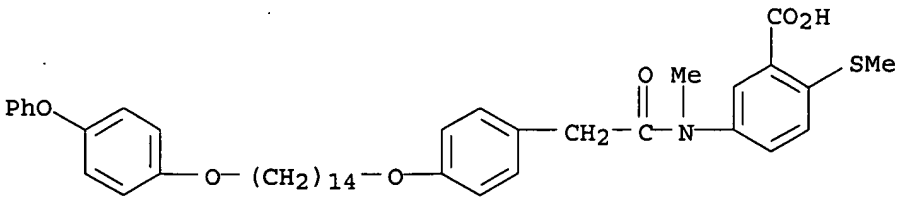
  6.8% PROCESSED    1000 ITERATIONS                                1 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS:  ONLINE  **COMPLETE**
                        BATCH   **COMPLETE**
PROJECTED ITERATIONS:       285919 TO   300401
PROJECTED ANSWERS:              64 TO      522

L2            1 SEA SSS SAM L1

=> d scan

L2    1 ANSWERS   REGISTRY  COPYRIGHT 2003 ACS
IN    Benzoic acid, 5-[methyl[[4-[[14-(4-phenoxyphenoxy)tetradecyl]oxy]phenyl]ac
      etyl]amino]-2-(methylthio)- (9CI)
MF    C43 H53 N O6 S
```



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

ALL ANSWERS HAVE BEEN SCANNED

=> s l1 ful
FULL SEARCH INITIATED 16:25:10 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 293378 TO ITERATE

100.0% PROCESSED 293378 ITERATIONS 1395 ANSWERS
SEARCH TIME: 00.00.06

L3 1395 SEA SSS FUL L1

=>
Uploading 09844061.str

L4 STRUCTURE UPLOADED

=> d l4
L4 HAS NO ANSWERS
L4 STR

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

Structure attributes must be viewed using STN Express query preparation.

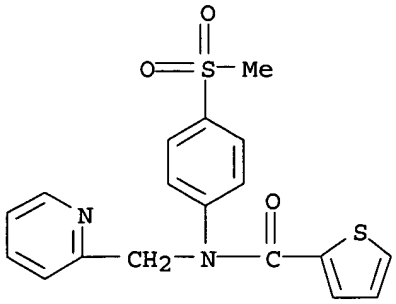
=> s l4 ful sub=l3
FULL SUBSET SEARCH INITIATED 16:26:10 FILE 'REGISTRY'
FULL SUBSET SCREEN SEARCH COMPLETED - 901 TO ITERATE

100.0% PROCESSED 901 ITERATIONS 900 ANSWERS
SEARCH TIME: 00.00.01

L5 900 SEA SUB=L3 SSS FUL L4

=> d scan

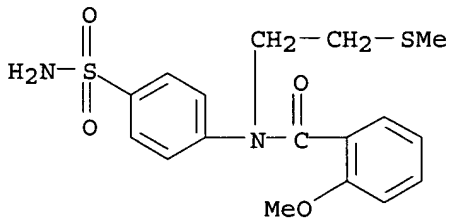
L5 900 ANSWERS REGISTRY COPYRIGHT 2003 ACS
IN 2-Thiophenecarboxamide, N-[4-(methylsulfonyl)phenyl]-N-(2-pyridinylmethyl)-
(9CI)
MF C18 H16 N2 O3 S2



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

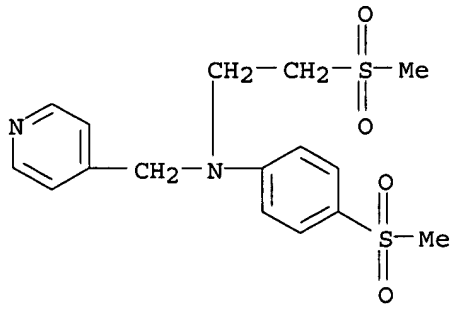
HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):2

L5 900 ANSWERS REGISTRY COPYRIGHT 2003 ACS
IN Benzamide, N-[4-(aminosulfonyl)phenyl]-2-methoxy-N-[2-(methylthio)ethyl]-(9CI)
MF C17 H20 N2 O4 S2



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L5 900 ANSWERS REGISTRY COPYRIGHT 2003 ACS
IN 4-Pyridinemethanamine, N-[2-(methylsulfonyl)ethyl]-N-[4-(methylsulfonyl)phenyl]- (9CI)
MF C16 H20 N2 O4 S2



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):end

=> d scanfil caplus
'SCANFIL' IS NOT A VALID FORMAT FOR FILE 'REGISTRY'
'CAPLUS' IS NOT A VALID FORMAT FOR FILE 'REGISTRY'

The following are valid formats:

Substance information can be displayed by requesting individual fields or predefined formats. The predefined substance formats are: (RN = CAS Registry Number)

REG - RN
SAM - Index Name, MF, and structure - no RN
FIDE - All substance data, except sequence data
IDE - FIDE, but only 50 names
SQIDE - IDE, plus sequence data
SQIDE3 - Same as SQIDE, but 3-letter amino acid codes are used
SQD - Protein sequence data, includes RN
SQD3 - Same as SQD, but 3-letter amino acid codes are used
SQN - Protein sequence name information, includes RN

CALC - Table of calculated properties
EPROP - Table of experimental properties
PROP - EPROP and CALC

Any CA File format may be combined with any substance format to obtain CA references citing the substance. The substance formats must be cited first. The CA File predefined formats are:

ABS -- Abstract
APPS -- Application and Priority Information
BIB -- CA Accession Number, plus Bibliographic Data
CAN -- CA Accession Number
CBIB -- CA Accession Number, plus Bibliographic Data (compressed)
IND -- Index Data
IPC -- International Patent Classification
PATS -- PI, SO
STD -- BIB, IPC, and NCL

IABS --ABS, indented, with text labels
IBIB -- BIB, indented, with text labels
ISTD -- STD format, indented

OBIB -------- AN, plus Bibliographic Data (original)
OIBIB ------- OBIB, indented with text labels

SBIB -------- BIB, no citations
SIBIB ------- IBIB, no citations

The ALL format gives FIDE BIB ABS IND RE, plus sequence data when it is available.
The MAX format is the same as ALL.
The IALL format is the same as ALL with BIB ABS and IND indented, with text labels.

For additional information, please consult the following help messages:

HELP DFIELDS -- To see a complete list of individual display fields.
HELP FORMATS -- To see detailed descriptions of the predefined formats.
ENTER DISPLAY FORMAT (IDE):end

=> fil caplus

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	186.25	186.46

FILE 'CAPLUS' ENTERED AT 16:29:34 ON 18 FEB 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

FILE COVERS 1907 - 18 Feb 2003 VOL 138 ISS 8
FILE LAST UPDATED: 17 Feb 2003 (20030217/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l5
L6 348 L5

=> d abs ibib hitstr 338-348

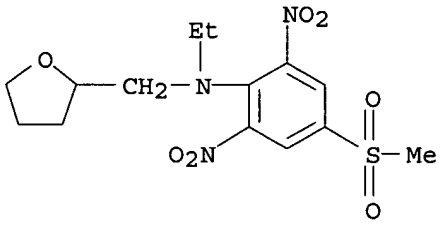
L6 ANSWER 338 OF 348 CAPLUS COPYRIGHT 2003 ACS
GI For diagram(s), see printed CA Issue.
AB Furfurylamine derivs. (I, R = Me, Cl, CN, F3C, F2CH, MeSO2, H2NCO; R1 = Me, Et, Pr, Bu, Me2CH, MeOCH2CH2; R2 = H, Me) and tetrahydrofurfuryl derivs. of I (II, R = Me, Cl, I, F3C, F2CH, MeSO2; R1 = Me, Et, Pr, Bu; R2 = H, Me) useful as pre- and postemergent herbicides for graminaceous weeds were prepd. by arylating the amines with 4,3,5-Cl(O2N)2C6H2R. Thus 56 g 4,3,5-Cl(O2N)2C6H2SO2Me, 27 g N-ethyltetrahydro-furfurylamine, and 21 g Et3N in EtOH gave 69 g II (R = MeSO2, R1 = Et, R2 = H). I (R = F3C; R1 = Me, Et, R2 = H) at preemergent applications of 1 and 8 kg/ha killed Digitaria sanguinalis, Poa trivialis, Alopecurus myosuroides, Echinochloa curs-galli and Setaria italica in cotton and soybeans with greater selectivity than 4-trifluoromethyl-2,6-dinitro-N,N-dipropylaniline.
ACCESSION NUMBER: 1972:488282 CAPLUS
DOCUMENT NUMBER: 77:88282
TITLE: Herbicidal furfurylamines
INVENTOR(S): Bader, Joerg; Schempp, Heinrich; Vogel, Christian
PATENT ASSIGNEE(S): Agripat S. A.
SOURCE: S. African, 30 pp.

CODEN: SFXXAB
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

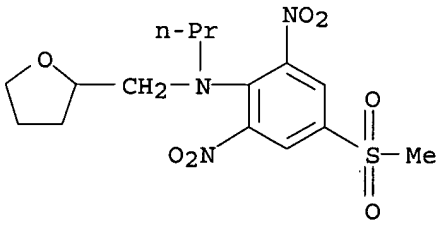
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ZA 7103898		19711214		
PRIORITY APPLN. INFO.:			CH 1970-9181	19700616

IT **34129-04-9P 34129-16-3P 38105-67-8P**
38105-69-0P 38105-77-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)

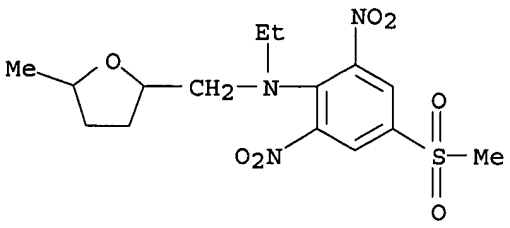
RN 34129-04-9 CAPLUS
CN 2-Furanmethanamine, N-ethyltetrahydro-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]- (9CI) (CA INDEX NAME)



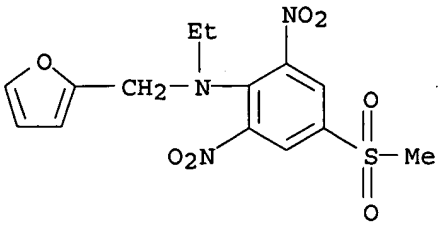
RN 34129-16-3 CAPLUS
CN 2-Furanmethanamine, tetrahydro-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]-N-propyl- (9CI) (CA INDEX NAME)



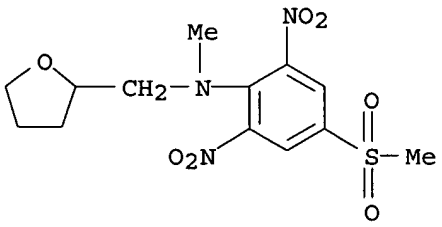
RN 38105-67-8 CAPLUS
CN 2-Furanmethanamine, N-ethyltetrahydro-5-methyl-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]- (9CI) (CA INDEX NAME)



RN 38105-69-0 CAPLUS
CN 2-Furanmethanamine, N-ethyl-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]- (9CI) (CA INDEX NAME)



RN 38105-77-0 CAPLUS
CN 2-Furanmethanamine, tetrahydro-N-methyl-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]- (9CI) (CA INDEX NAME)



L6 ANSWER 339 OF 348 CAPLUS COPYRIGHT 2003 ACS
AB The glycidylamides I (A = n-valent radical, n > 1, R = monovalent radical, X = H or Me), useful in the prepn. of epoxy resins, are prepd. by reaction of the corresponding amides with epihalohydrins. Thus, a mixt. of bis(4-acetamidophenyl) sulfone 166.2, epichlorohydrin 1387.5, and 50% Me4NCl 5.9 g is refluxed 2 hr, vacuum distd. at 60.deg./60-95 mm, and treated over 3.5 hr with 100.0 g 50% NaOH to give 178.0 g N,N'-diacetyl-N,N'-diglycidylbis(p-aminophenyl) sulfone (I, A = sulfonyl-di-p-phenylene, n = 2, R = Me, X = H) (II) [**35187-00-9**]. The exothermic reaction of II 35, 1,4-butanediol diglycidyl ether 25, and hexahydrophthalic anhydride 52 parts gives a red resin having good mech. properties.

ACCESSION NUMBER: 1972:435489 CAPLUS
DOCUMENT NUMBER: 77:35489
TITLE: Polyglycidyl compounds useful in hardenable epoxy resin mixtures
INVENTOR(S): Habermeier, Juergen; Batzer, Hans; Porret, Daniel
PATENT ASSIGNEE(S): Ciba-Geigy A.-G.
SOURCE: Ger. Offen., 39 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2148409	A	19720330	DE 1971-2148409	19710928

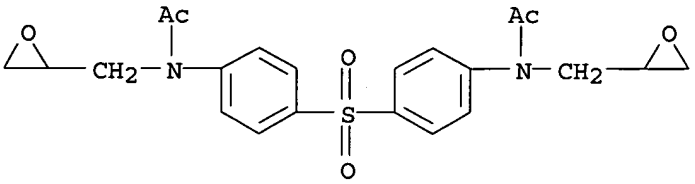
CH 541552	A	19731031	CH 1970-14503	19700929
GB 1360264	A	19740717	GB 1971-44959	19710927
FR 2108063	A1	19720512	FR 1971-34812	19710928
FR 2108063	A5	19720512		

PRIORITY APPLN. INFO.: CH 1970-14503 19700929

IT **35187-00-9P**
RL: PREP (Preparation)
(manuf. of, for epoxy resin prepn.)

RN 35187-00-9 CAPLUS

CN Acetamide, N,N'-(sulfonyldi-4,1-phenylene)bis[N-(oxiranylmethyl)- (9CI) (CA INDEX NAME)



L6 ANSWER 340 OF 348 CAPLUS COPYRIGHT 2003 ACS

AB Herbicidal title compds. 4,2,6-R2(O2N)2C6H2NRR1 (I) were prepd. by reaction of 4,2,6-R2(O2N)2C6H2Cl (II) with HNRR1 and used at 5.60 and 11.2 kg/ha, resp., in pre- and post-emergence tests as suspensions against weeds, e.g. crabgrass, zinnia, or foxtail, without affecting, e.g., cotton, corn, or rice. Thus, 5.5 g II (R2=CF3) in 30 ml C6H6 was added within 20 min to a mixt. contg. 3.3 g N-propyltetrahydrofurfurylamine and 3.0 g Et3N in 125 ml C6H6 and the mixt. refluxed 5 hr to give 6.8 g red oily I (R=Pr, R1=tetrahydrofurfuryl, R2=CF3). Similarly prepd. were 26 other I, e.g. (R-R2 given): Pr, CH2Ph, CF3; Et, 2-picolyl, SO2Me; and Pr, CH2Ph, SO2NH2.

ACCESSION NUMBER: 1971:551512 CAPLUS
DOCUMENT NUMBER: 75:151512
TITLE: Herbicidal 2,6-dinitroanilines
INVENTOR(S): Maravetz, Lester L.
PATENT ASSIGNEE(S): Esso Research and Engineering Co.
SOURCE: Ger. Offen., 37 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

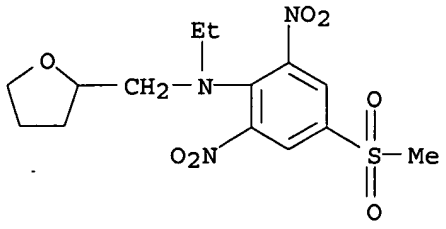
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2108346	A	19710916	DE 1971-2108346	19710222
US 3686230	A	19720822	US 1970-18407	19700224

PRIORITY APPLN. INFO.: US 1970-18407 19700224

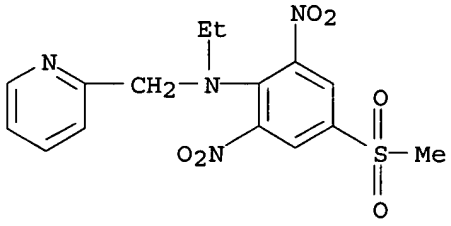
IT **34129-04-9P 34129-05-0P 34129-15-2P 34129-16-3P 34129-21-0P 34129-22-1P 34129-23-2P 34129-24-3P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)

RN 34129-04-9 CAPLUS

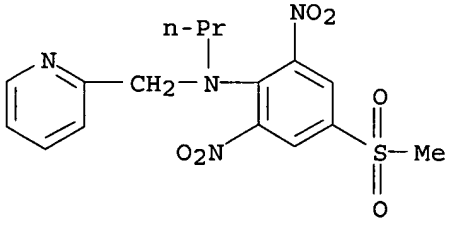
CN 2-Furanmethanamine, N-ethyltetrahydro-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]- (9CI) (CA INDEX NAME)



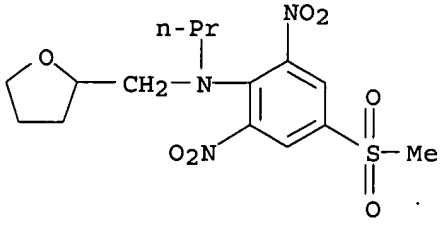
RN 34129-05-0 CAPLUS
CN 2-Pyridinemethanamine, N-ethyl-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]-(9CI) (CA INDEX NAME)



RN 34129-15-2 CAPLUS
CN 2-Pyridinemethanamine, N-[4-(methylsulfonyl)-2,6-dinitrophenyl]-N-propyl-(9CI) (CA INDEX NAME)

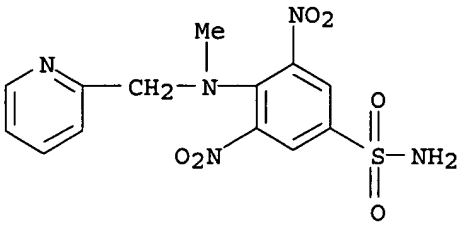


RN 34129-16-3 CAPLUS
CN 2-Furanmethanamine, tetrahydro-N-[4-(methylsulfonyl)-2,6-dinitrophenyl]-N-propyl- (9CI) (CA INDEX NAME)

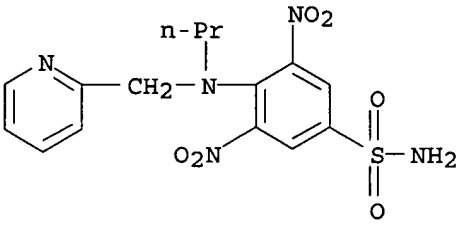


RN 34129-21-0 CAPLUS
CN Benzenesulfonamide, 4-[methyl(2-pyridinylmethyl)amino]-3,5-dinitro- (9CI)

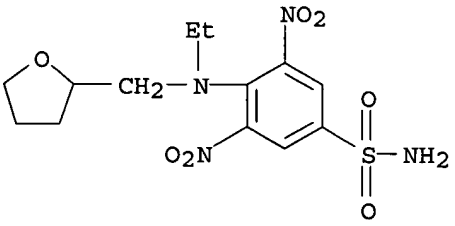
(CA INDEX NAME)



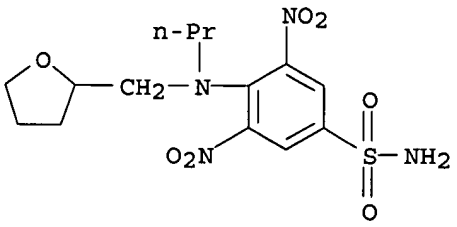
RN 34129-22-1 CAPLUS
CN Benzenesulfonamide, 3,5-dinitro-4-[propyl(2-pyridinylmethyl)amino]- (9CI) (CA INDEX NAME)



RN 34129-23-2 CAPLUS
CN Benzenesulfonamide, 4-[ethyl[(tetrahydro-2-furanyl)methyl]amino]-3,5-dinitro- (9CI) (CA INDEX NAME)



RN 34129-24-3 CAPLUS
CN Benzenesulfonamide, 3,5-dinitro-4-[propyl[(tetrahydro-2-furanyl)methyl]amino]- (9CI) (CA INDEX NAME)



L6 ANSWER 341 OF 348 CAPLUS COPYRIGHT 2003 ACS
GI For diagram(s), see printed CA Issue.
AB The title compds. of the general formula I, where Z is the residue of an aminoazo compds. contg. .gtoreq.2 SO3H groups, optionally copperized, and X = O or SO2, are yellowish orange to blue dyes for cellulosic textiles. Thus, (p-H2NC6H4)2SO2 and II yielded the corresponding bluish red I. Similarly, 7 other I were prepd.

ACCESSION NUMBER: 1970:500001 CAPLUS
DOCUMENT NUMBER: 73:100001
TITLE: Fiber-reactive dyes
INVENTOR(S): Andrew, Herbert F.
PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.
SOURCE: Ger. Offen., 38 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

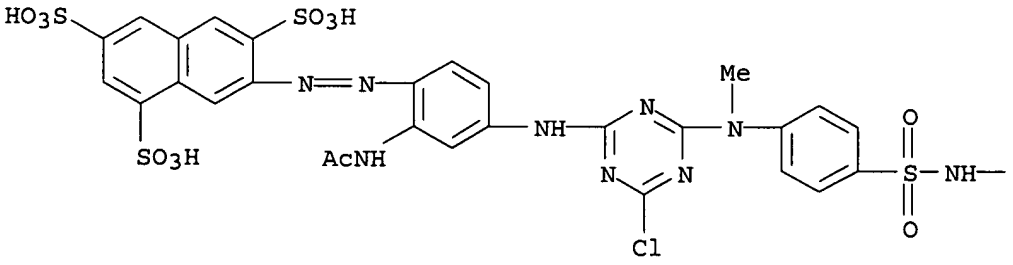
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2000518	A	19700716	DE 1970-2000518	19700107
GB 1260582	A	19720119	GB 1969-1037	19690107
BR 7015829	A0	19730419	BR 1970-215829	19700106
NL 7000136	A	19700709	NL 1970-136	19700107
FR 2027883	A5	19701002	FR 1970-452	19700107

PRIORITY APPLN. INFO.: GB 1969-1037 19690107

IT **29330-69-6 29399-38-0**
RL: USES (Uses)
(fixation on fiber)

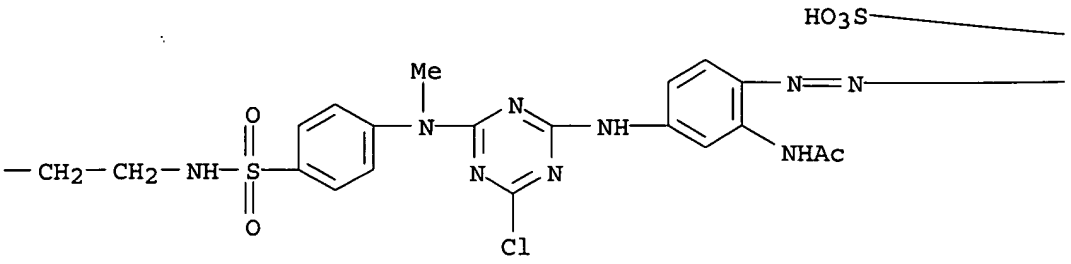
RN 29330-69-6 CAPLUS
CN 1,3,6-Naphthalenetrisulfonic acid, 7,7'-[ethylenebis[iminosulfonyl-p-phenylene(methylimino)(6-chloro-s-triazine-4,2-diyl)imino(2-acetamido-p-phenylene)azo]]di-, hexasodium salt (8CI) (CA INDEX NAME)

PAGE 1-A

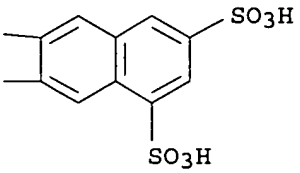


●6 Na

PAGE 1-B

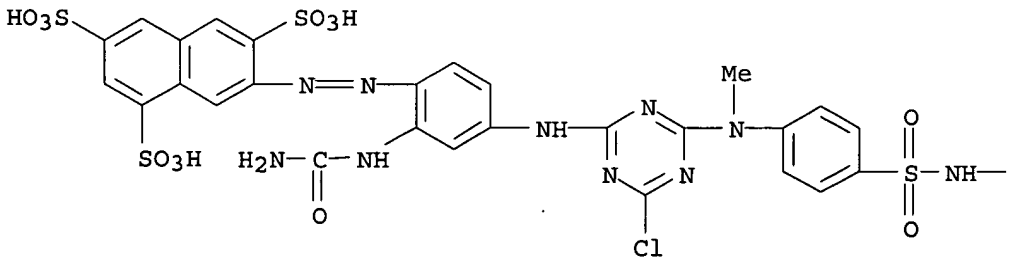


PAGE 1-C



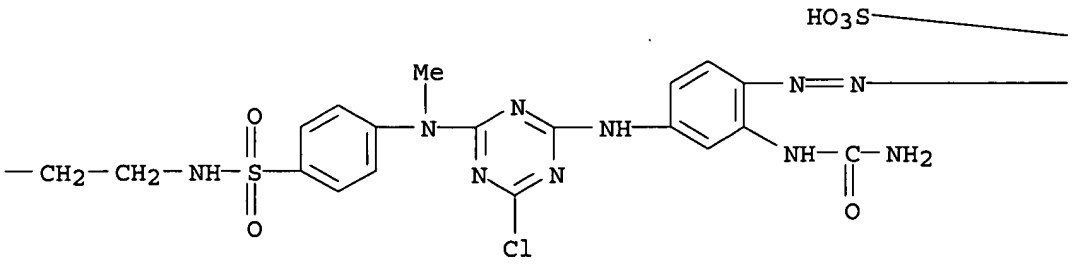
RN 29399-38-0 CAPLUS
CN 1,3,6-Naphthalenetrisulfonic acid, 7,7'-[ethylenebis[iminosulfonyl-p-phenylene(methylimino)(6-chloro-s-triazine-4,2-diyl)imino(2-ureido-p-phenylene)azo]]di-, hexasodium salt (8CI) (CA INDEX NAME)

PAGE 1-A

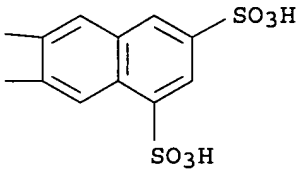


●6 Na

PAGE 1-B



PAGE 1-C



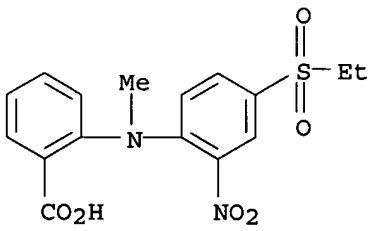
L6 ANSWER 342 OF 348 CAPLUS COPYRIGHT 2003 ACS

AB A mixt. of 57.5 g. 2-nitro-4-ethylsulfonylchlorobenzene, 31.5 g. anthranilic acid, and 300 ml. n-C5H11OH is refluxed 2 hrs. with 33 g. K2CO3 and 1 g. Cu powder to give 55 g. 4,2-EtSO2(O2N)-C6H3NRC6H4CO2H-2 (I) (R = H), m. 244-5.degree. (EtOH-Me2CO). Similarly is prepd. I (R = Me), m. 171-2.degree. (iso-PrOH-C6H6). I (R = H) (55 g.) is heated with 1.6 l. MeOH satd. with HCl, the resulting Me ester (m. 134-6.degree.) subjected to catalytic redn. using 25 ml. Raney Ni in 750 ml. MeOH, and the resulting Me N-[2-amino-4-(ethylsulfonylphenyl)]anthranilate (m. 128-30.degree.) heated with 20% H2SO4 4 hrs. to give 84.2% II (R = H), m. 141-3.degree. (EtOH-Me2CO). Also is prepd. II (R = Me), m. 256-7.degree. (dioxane). II (R = H) (3 g.) in 100 ml. dioxane is refluxed 3 hrs. with 0.6 g. NaNH2 and heated 2.5 hrs. with 1.1 g. Me2N(CH2)2Cl to give 2.61 g. III (R = H, n = 2), m. 163-5.degree.. Similarly prepd. are the following III (R, n, % yield, and m.p. given): H, 3, 54, 230-2.degree.; Me, 2, 78, 217-19.degree.; and Me, 3, 77, 231-2.degree.. III (R = H, n = 2) (3 g.) is heated with 1.2 g. LiAlH4 and 80 ml. 3:1 tetrahydrofuran-Et2O 40 hrs. to give 0.97 g. IV (R = H), m. 202-7.degree. (MeOH-Et2O). Similarly prepd. is IV (R = Me), m. 200-2.degree. (EtOH-Et2O). II (R = H) (10 g.) is refluxed with 1.95 g. NaNH2 and 5 g. MeI in dioxane to give 6.8 g. 8-ethylsulfonyl-10-methyl-5H-dibenzo[b,e][1,4]diazepin-11(10H)-one (V), m. 151-61.degree. (EtOH). V (5 g.) in 100 ml. xylene is refluxed 3 hrs. with 0.95 g. 43.9% NaH and heated another 17 hrs. with 1.8 g. Me2N(CH2)2Cl to give 3.03 g. 5-(2-dimethylaminoethyl)-8-ethylsulfonyl-10-methyl-5H-dibenzo[b,e] [1,4]diazepin - 11(10H)-one-HCl, m. 265-9.degree. (MeOH-Et2O). 2,4-Dinitrochlorobenzene (33.3 g.) is condensed with 23.3 g. anthranalic acid (VI) by refluxing 6 hrs. in a mixt. of 27 g. K2CO3, 0.5 g. KI, and 600 ml. EtOH to give 42.7 g. VII (R = H), m. 258-9.5.degree. (EtOH-Me2CO). The use of N-methylanthranilic acid instead of VI gives VII (R = Me), m. 176-8.degree. (EtOH). VII (R = H) (50 g.) is heated with 39 g. PCl5 in 700 ml. C6H6 6 hrs., the resulting acid chloride heated in 700 ml. MeOH 3.5 hrs., the Me ester (45 g.) (m. 166-7.degree.) thus obtained

subjected to catalytic redn. over Raney Ni in 700 ml. MeOH, and the resulting amino compd. (m. 126-9.degree.) (2 g.) refluxed 4 hrs. with 0.7 g. NaNH2 and 30 ml. dioxane to give 0.44 g. VIII (R = H), m. 215-16.degree. (MeOH). VII (R = Me) (75 g.) is subjected to catalytic redn. over Raney Ni in 550 ml. MeOH with 110 atm. H, and the resulting powder (m. 155-60.degree.) heated with 500 ml. 10% HCl 45 min. and worked up to give 10.5 g. VIII (R = Me), m. 233-5.degree. (EtOH-Me2CO). VIII (R = Me) (3 g.) is diazotized with 0.9 g. NaNO2 in a mixt. of 7 ml. concd. HCl and 5 ml. AcOH and treated with 10 ml. 40% NHMe2 soln. to give 1.25 g. 5-methyl-8-dimethylsulfamyl-5H-dibenzo-[b,e][1,4]diazepin-11(10H)-one (IX), m. 266-9.degree. (aq. dioxane). IX is treated as in the prepn. of III to give 37.5% 5-methyl-8-dimethylsulfamyl-10-(2-dimethylaminoethyl)-5H - dibenzo [b,e]-[1,4]diazepin-11(10H)-one; HCl salt m. 192-5.degree. (EtOH-Me2CO). Among the compds. synthesized, III (R = Me, n = 2) was found to be a potent antidepressant with low toxicity.

ACCESSION NUMBER: 1969:403368 CAPLUS
DOCUMENT NUMBER: 71:3368
TITLE: Synthesis of dibenzo[b,e][1,4]diazepine derivatives as anti-depressants
AUTHOR(S): Takeda, Mikio; Matsubara, Mitsuru; Kugita, Hiroshi
CORPORATE SOURCE: Org. Chem. Re. Lab., Tanabe Seiyaku Co., Ltd., Toda, Japan
SOURCE: Yakugaku Zasshi (1969), 89(2), 158-63
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Japanese

IT **22777-14-6P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
RN 22777-14-6 CAPLUS
CN Anthranilic acid, N-[4-(ethylsulfonyl)-2-nitrophenyl]-N-methyl- (8CI) (CA INDEX NAME)



L6 ANSWER 343 OF 348 CAPLUS COPYRIGHT 2003 ACS
GI For diagram(s), see printed CA Issue.
AB 2,4-Dihalo-5-sulfamoylbenzoic acids and their functional derivs. reacted at higher temp. with primary and secondary amines, NH3, and N2H4 with the exchange of 1 halogen atom by a basic group. Some of the condensation products, particularly 4-chloro-5-sulfamoyl-N-(2-furylmethyl)anthranilic acid (furosemide) (I), exhibited a high saluretic and diuretic activity. 2,4-Cl2C6H3CO2H (40 g.) added at room temp. with stirring to 120 cc. ClSO3H, heated rapidly to 155.degree. stirred 2 hrs. at 155.degree., cooled, and added dropwise to 1 kg. ice, and the moist, yellowish 2,4,5-Cl2(ClO2S)C6H2CO2H (II) [dried, m. 167-75.degree., 184.degree. (CHCl3-petr. ether)] added in portions with stirring and cooling to 400 cc. concd. HCl, kept overnight, and acidified with HCl to pH 2 yielded 39 g. III (R = R' = H, X = Y = Cl) (IV), m. 233.degree. (H2O). II with 400

cc. 10% aq. MeNH2 gave similarly 34 g. III (R = Me, R' = H, X = Y = Cl) (V), m. 200.degree. (50% EtOH), and with 400 cc. 15% aq. Me2NH yielded 39 g. III (R = R' = Me, X = Y = Cl), m. 182.degree. (aq. EtOH). 2,4-H2NClC6H3CO2Et (100 g.) and 300 cc. 5N HCl heated 10 min. on a steam bath, cooled to 0.degree. treated with 35 g. NaNO2, filtered, and treated 1 hr. at 0.degree. with 200 g. 60% HBF4 yielded 116 g. [5,2-Cl(EtO2C)C6H3N2]BF4, decomp. 147.degree., which fused over a free flame until the BF3 evolution ceased gave crude 4,2-ClFC6H3CO2Et; this refluxed 1 hr. with 40 g. KOH in 200 cc. 50% EtOH and acidified with 2N HCl yielded 48 g. 4,2-ClFC6H3CO2H (VI), m. 203-4.degree. (30% EtOH). VI (35 g.) treated successively with ClSO3H and NH4OH gave 26g. III (R = R' = H, X = Cl, Y = F) (VII), m. 242-3.degree. (80% EtOH). 4,2-H2NClC6H3CO2Et (100 g.) was converted via the [3,4-Cl(EtO2C)C5H3N2]BF4, decomp. 125.degree., and 2,4-ClFC6H3CO2H, m. 180-1.degree., to 27 g. (crude) III (R = R' = H, X = F, Y = Cl) (VIII), m. 246.degree. (H2O). IV (27 g.) refluxed 1 hr. with 35 cc. SOCl2 and evapd., and the residue dissolved in 100 cc. MeOH, basified dropwise with cooling with Et3N, and warmed to room temp. yielded 21.6 g. Me ester of IV, m. 202.degree. (80% EtOH). Similarly was prepd. the Et ester of IV, 77%, m. 116.degree. (EtOH). IV (27 g.) treated with SOCl2, and the crude acid chloride stirred into 200 cc. concd. NH4OH, concd. to half-vol., and adjusted to pH 4.0 gave 16 g. amide (IX) of IV, m. 208-10.degree. (80% EtOH). The acid chloride from a similar run treated with 100 cc. 40% aq. EtNH2 gave 21 g. ethylamide (X) of IV, m. 214.degree. (EtOH). A similar run with 40 cc. BuNH2 in 100 cc. 80% tetrahydrofuran (THF) gave 23 g. butylamide (XI) of IV, m. 180.degree. (90% EtOH). VIII (25.3 g.) in 250 cc. MeOH treated with 1.05 equiv. CH2N2Et2O and kept briefly at room temp. yielded 23.5 g. Me ester of VIII, m. 163-4.degree.. 2,4-Br2C6H3CO2H (56 g.) treated successively with ClSO3H and NH4OH yielded 36 g. III (R = R' = H, X = Y = Br) (XII), m. 243.degree. (aq. HCONMe2). The appropriate III heated with 3-10 equivs. amine with or without solvent heated to a predetd. temp. (runs at temps. above the b.p. of the solvent were performed in an autoclave under N), and the mixt. poured into dil. HCl gave the corresponding XIII. VI (25.3 g.) in 50 g. furfurylamine (XIV) heated 2 hrs. at 95.degree., dild. with 500 cc. H2O, and acidified at 0.degree. with AcOH gave 28 g. I, decomp. 208.degree. (aq. EtOH). IV (50 g.) and 100 g. XIV heated 4 hrs. at 130.degree. and stirred into 1 l. cold 10% AcOH gave 26 g. I, decomp. 205.degree. (above 245.degree. with blackening). I (1.0 g.) in 10 cc. N NaOH heated 1 hr. on the steam bath and acidified with AcOH was recovered unchanged. I (3.3 g.) and 50 cc. N HCl refluxed 1 hr. gave 0.4 g. III(R = R' = H, X = Cl, Y = NH2), decomp. 265.degree. (aq. EtOH). I (66.2 g.) in 600 cc. THF treated with 41.2 g. dicyclohexylcarbodiimide and kept 1 day at room temp. in the dark, and the crude product extd. with 800 cc. boiling EtOH left cryst. anhydride of I and gave 11 g. N-[4-chloro-5-sulfamoyl-2-(2-furylmethylamino)benzoyl]-N,N'-dicyclohexylurea (XV), m. 163-5.degree.. The insol. anhydride dissolved in 200 cc. warm HCONMe2, filtered, dild. with 200 cc. H2O in portions, and kept 3 hrs. at room temp. gave 38 g. pure, pale yellow anhydride (XVI) of I, decomp. 183-5.degree.. XVI (7.0 g.) in 70 cc. 2N NaOH kept 2 hrs. at room temp. and adjusted with 2N HCl to pH 2 yielded 4.8 g. cryst. solid, presumably XVII, decomp. above 210.degree. with blackening. XVI (1 g.) and 10 cc. 20% NH4OH stirred 15 min. at 80.degree. gave the amide (XVIII) of I, m. 217.degree. (aq. HCONMe2); the aq. filtrate acidified yielded I. Me ester (XIX) (6.9 g.) of I in 50 cc. dioxane heated at 90.degree. with 3.0 g. LiAlH4 gave 2.8 g. pale yellow 4-chloro-5-sulfamoyl-2-(2-furylmethylamino)benzyl alc., m. 157.degree. (H2O). I (25 g.), 25 cc. Ac2O, and 100 cc. C5H5N heated 1 hr. on the steam bath, dild. with 500 cc. H2O, and acidified with 3N HCl to pH 3.0 gave 24.2 g. diacetyl deriv. of I, decomp. 205-6.degree. (EtOH). I (16.5 g.) and 7.6 cc. Et3N in 100 cc.

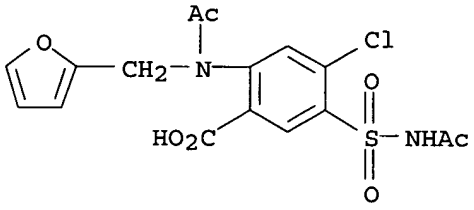
dry THF treated at -5.degree. with stirring with 5.2 cc. ClCO2Et, stirred 5 min. at 0.degree., and poured into 100 cc. cold, concd. NH4OH yielded 2.4 g. XVIII, decomp. 223.degree. (aq. HCONMe2). XVIII (4.0 g.) in 40 cc. N NaOH refluxed 1 hr., dild. with H2O, and adjusted with AcOH to pH 8.0 gave 1.9 g. I, decomp. 204-5.degree.. XI (9.8 g.) and 20 cc. XIV heated 3 hrs. on the steam bath gave 8.5 g. butylamide of I, m. 180-1.degree. (EtOH). XVI (6.5 g.) in 30 cc. THF treated 0.5 hr. at room temp. with 30 cc. PhCH2NH2 gave 3.9 g. benzylamide of I, m. 195-7.degree. with yellowing (EtOH). Similarly was prepd. 1.3 g. N,N-pentamethylenehydrazide of I, m. 196-7.degree. (70% EtOH), from 3.0 g. N,N-pentamethylenehydrazine. H2NCH2CO2Et (3.0 g.) with XVI gave 2.2 g. N-carbethoxymethylamide of I, m. 176.degree. (EtOH), which treated 1 hr. at 25.degree. with 15 cc. N NaOH and adjusted with N HCl to pH 3 yielded 1.7 g. N-carboxymethylamide of I, decomp. 203.degree.. I (33 g.) in 100 cc. THF treated 5 min. with about 200 cc. CH2N2-Et2O yielded 25 g. XIX, m. 184-6.degree.. IV (8.9 g.) in 25 cc. XIV heated 1 hr. at 90.degree. and treated with 200 cc. 10% AcOH yielded 10.6 g. (crude) Et ester (XX) of I, m. 165-7.degree. (EtOH). XX (0.1 g.) in 2 cc. 2N NaOH heated 10 min. at 70.degree. and treated with AcOH gave I. IV (8.9 g.) and 25 cc. XIV heated 2 hrs. at 115.degree. and poured into dil. AcOH, and the ppt. (4.6 g.), m. 134-6.degree. warmed briefly with 30 cc. 2N NaOH at 60-70.degree. and adjusted with AcOH to pH 5 yielded 2.5 g. 4-(2-furylmethylamino)-5-sulfamoyl-N-(2-furylmethyl)-anthranilic acid, decomp. 217.degree. (EtOH). XVIII (3.3 g.), 40 cc. EtOH, 2.0 cc. N NaOH, and 1.2 g. 30/% aq. CH2O refluxed 0.5 hr. gave 2.3 g. 7-chloro-6-sulfamoyl-1-(2-furylmethyl)-4-oxo-1,2,3,4-tetrahydroquinazoline, decomp. 245.degree. (aq.HCONMe2). XII (18 g.) and 36 g. XIV heated 2 hrs. at 125.degree. gave 3.4 g. XIII (R = R' = R'' = H, R''' = 2-furylmethyl, X = Br), decomp. 216.degree. (EtOH). VII (8.9 g.) and 20 cc. PhCH2NH2 heated 1.5 hrs. on a steam bath and poured into 250 cc. 10% AcOH, and the ppt. repptd. from 250 cc. N NaHCO3 with 2N HCl yielded 11.8 g. XIII (R = R' = R'' = H, R''' = PhCH2, X = Cl)(XXI), decomp. 244.degree. (EtOH). IV (27 g.) and 42 cc. PhCH2NH2 in MeOCH2CH2OH refluxed 3 hrs. yielded 16 g. XXI, decomp. 244-5.degree. (EtOH). Similarly were prepd. the XIII (X = Cl) listed in the 1st table. XII (36 g.) and PhCH2NH2 gave similarly during 3 hrs. 19 g. XIII (R = R' = R'' = H, R''' = PhCH2, X = Br), decomp. 247.degree. (50% EtOH). R, R', R'', R''', m.p., % yield, reflux time (hrs.); H, Me, H, PhCH2, 238.degree., 70, 3; Me, Me, H, PhCH2, 206.degree., 27, 3; H, H, H, .omicron.-MeOC6H4CH2, 220.degree., 27, 4; H, H, H, p-MeC6H4CH2, 230-1.degree., 35, 4; H, H, Me, PhCH2, 202.degree. (decompn.), 42, 2; H, H, H, 2-thenylmethyl, 201.degree. (decompn.), 87, 3; H, H, H, iso-Bu, 236.degree., 46, 3; H, H, H, MeO(CH2)3, 204.degree., 35, 3; XXI (68.2 g.) with dicyclohexylcarbodiimide yielded 41 g. pale yellow anhydride of XXI, decomp. 207.degree. (repptd. from HCONMe2 with H2O). XXI (34.1 g.) in 100 cc. dioxane treated dropwise with stirring at 80.degree. with 20.0 cc. SOCl2, stirred 15 min. at 80.degree., and dild. with 300 cc. petr. ether, and the resulting acid chloride added in portions with stirring and cooling to 150 cc. THF and 200 cc. concd. NH4OH yielded 23.0 g. amide of XXI, m. 224.degree. (HCONMe2-H2O). X (18.0 g.) in 40 cc. PhCH2NH2 heated 2 hrs. at 110.degree. and poured into 200 cc. 2N HCl yielded the ethylamide of XXI, m. 251-2.degree. (HCONMe2-H2O). XXI (3.4 g.) condensed with CH2O gave 3.0 g. 7-chloro-6-sulfamoyl-1-benzyl-4-oxo-1,2,3,4-tetrahydroquinazoline (XXII), m. 244-5.degree. (decompn.) (HCONMe2-H2O). XIII (3.0 g.) in 60 cc. HCONMe2 hydrogenated under ambient conditions 10 min. over Pd black gave 1.9 g. 7-chloro-6-sulfamoyl-1,2,3,4-tetrahydroquinazoline, m. 256-8.degree. (decompn.). XXI (10 g.) in 200 cc. MeOH satd. a room temp. with dry HCl and kept overnight gave 5.2 g. Me ester of XXI, m. 188.degree. (aq. HCONMe2). Me ester (26.7 g.) of VI and 100 g. (PhCH2)2NH heated 3 hrs. on a steam bath and stirred into 1 l. N

AcOH, and the ppt. heated 15 min. at 100.degree. with 500 cc. 0.5N NaOH gave 36.6 g. XIII (R = R' = H, R'' = R''' = PhCH2, X = Cl), decomp. 206.degree.. IV (5.4 g.) and 8 g. MePhCHNH2 in (CH2OH)2 heated 3 hrs. at 150.degree. yielded 0.5 g. XIII (R = R' = R'' = H, R''' = MePhCH, X = Cl), m. 191-3.degree. (aq. EtOH). IV (10.8 g.) and 25 cc. PhCH2CH2NH2 in (MeOCH2CH2)2O refluxed 2 hrs. and acidified with HCl gave 12.2 g. XIII (R = R' = R'' = H, R''' = PhCH2CH2, X = Cl), decompg. 215.degree. (50% EtOH); method A. IV (10 g.) and 30 cc. PhNH2 refluxed 12 hrs. and acidified with 200 cc. 2N HCl gave 5.7 g. XIII (R = R' = R'' = H, R''' = Ph, X = Cl), decomp. 245.degree. (40% MeOH); method B. IV (27 g.) and 200 cc. 10% aq. MeNH2 heated 5 hrs. at 125-30.degree. yielded 14 g. XIII (R = R' = R'' = H, R''' = Me, X = Cl), m. 264.degree. (decompn.) (35% EtOH); method C. IV (10.8 g.) and 16 cc. piperidine in BuOCH2CH2OH refluxed 3 hrs. gave 10.4 g. (crude) 4-chloro-5-sulfamoyl-N,N-pentamethylenanthranilic acid, decomp. 224.degree. (50% MeOH); method D. Similarly were prepd. the XIII listed in the 2nd table. VI (5.1 g.) and 6.3 g. 1-C10H7CH2NH2 in 15 cc. C5H5N refluxed 2 hrs., dild. with H2O, and acidified with HCl to pH 3 gave 6.3 g. XIII (R = R' = R'' = H, R''' = 1-C10H7CH2, X = Cl), decomp. 222-3.degree. (90% EtOH). Amide (XXV) (5.8 g.) of XXIII, m. 232-3.degree. (aq. HCONMe2) in 300 cc. AcOH treated dropwise at 50.degree. with 1.02 cc. Br in 30 cc. AcOH and dild. with 600 cc. H2O yielded 5.3 g. dibromide of XXV, decomp. 193.degree. (80% EtOH). XXIV (20 g.) in 60 cc. 5N NaOH heated 2 hrs. on the steam bath and adjusted with dil. HCl to pH 7 gave 12.7 g. XIII (R = R' = R'' = H, R''' = CH2CH2NH2, X = Cl), decomp. 269.degree.. IV (10.8 g.) and 7.5 g. 80% N2H4 refluxed 2 hrs. in 20 cc. MeOCH2CH2OH and poured into 200 cc. H2O gave 6.2 g. pale yellow XIII (R = R' = R'' = H, R''' = NH2, X = Cl) (XXVI), decomp. 290.degree. (aq. HCONMe2). XXVI (1.5 g.) recrystd. from boiling N HCl and then H2O gave 1.0 g. 6-chloro-3-oxo-5-sulfamoylindazoline, decomp. 290.degree.. VIII (8.9 g.) in 20 cc. PhCH2NH2 heated 3 hrs. on a steam bath gave 11.5 g. 2,4,5-Cl(PhCH2NH) (H2NO2S)C6H2CO2H (XXVII), decomp. 232.degree. (EtOH). IX (16.-2 g.) and 16.2 g. PhCH2NH2 in 60 cc. MeOCH2CH2OH refluxed 3 hrs. and poured into 300 cc. 5% AcOH, and the pptd. isomer mixt. (18.8 g,), m. 195-205% extd. twice with 250 cc. 90% boiling EtOH gave 1.6 g. amide (XXVIII) of XXVII, m. 260-2.degree. (aq. HCONMe2). XXVIII (3.4 g.), 1.0 cc. 30% aq. CH2O, 20 cc. EtOH, 20 cc. (MeOCH2CH22)2O, and 10 cc. 0.2N NaOH heated 1 hr. on a steam bath yielded 2.7 g. 6-chloro-7-carbamoyl-4-benzyl-2,3-dihydro-4H-1,2,4-benzothiadiazine 1,1-dioxide, m. 244.degree. (aq. HCONMe2). R, R', R'', R''', X, m.p., % yield (method), reaction time (hrs.); , H, H, H, Me, Cl, 242-4.degree. (decompn.), 66, (C), 2; H, H, H, cyclohexylmethyl, Cl, 213.degree., -- (A), 3; H, H, H, 2-tetrahydrofurylmethyl, Cl, 228.degree. (decompn.), -- (A), 3; H, H, H, cyclohexyl, Cl, 248-9.degree. (decompn.), 40 (A), 3; H, H, H, C8H17, Cl 211.degree., 43 (A), 3; H, H, H, CH2:CHCH2 (XXIII), Cl, 218.degree. (decompn.), 71 (C), 2; H, H, Et, Et, Cl, 214.degree., 50 (C), 5; H, H, H, EtSCH2CH2, Cl, 192-3.degree., 42 (A), 3; H, H, H, CH2CH2OH, Cl, 246.degree. (decompn.), 48 (B), 2; H, H, H, CH2CH2NHAc (XXIV), Cl, 249.degree. (decompn.), 57 (D), 3; H, H, H, H, Cl, 270-2.degree. (decompn.), 83 (C), 3; VIII (4.0 g.) in 12 cc. XIV heated 2 hrs. on a steam bath, poured into 120 cc. 5% AcOH, and adjusted with HCl to pH 3 gave 3.45 g. III (R = R' = H, X = 2-furylmethylamino, Y = Cl) (XIX), decomp. 201-2.degree. with blackening (50% EtOH). XXIX (10 g.) in 50 cc. anhyd. HCO2H refluxed 2 hrs. gave 6.9 g. XXX, decomp. 336-8.degree.. XXX (10 g.) in 120 cc. N NaHCO3 treated at room temp. with 4.0 g. NaBH4 and kept 1 hr. at room temp. gave 6.9 g. 2,3-dihydro deriv. of XXX, decomp. 235-7.degree.. XXX (5.2 g.) in 100 cc. 2N NaOH heated 2 hrs. on the steam bath with 100 cc. 2N NaOH, cooled, and adjusted with 5N HCl to pH 2 yielded 3.5 g. III (R = R' = H, X = NH2, Y = Cl), decomp. 232-3.degree., which with CH2N2-THF gave the Me ester, m. 225.degree.. VIII converted to

the amide, m. 221.degree., and then heated 2 hrs. on the steam bath with 4 parts XIV gave the amide of XXIX, m. 226-7.degree. (aq. EtOH). XXIX with CH2N2-THF gave the Me ester of XXIX, m. 137.degree. (AcOEt-petr. ether). XXIX (3.3 g.) in 50 cc. EtOH heated 1 hr. on a steam bath with 1.5 cc. aq. CH2O and 2 cc. N NaOH and treated with 150 cc. 1% AcOH yielded 2.9 g. 4-furylmethyl-2,3-dihydro-4H- analog of XXX, decomp. 223-4.degree. with blackening and gas evolution. VIII (4.0 g.) in 12.0 cc. 2-tetrahydrofurylmethylamine stirred 1 hr. at 110.degree. and poured into 80 cc. 2N HCl gave 2.7 g. XIII (R = R' = R'' = H, R''' = 2-tetrahydrofurylmethyl, X = Cl), m. 217-18.degree. (75% EtOH).

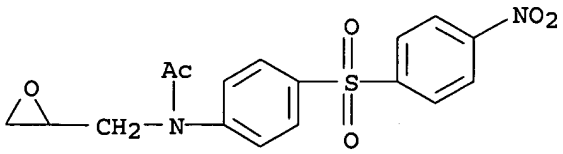
ACCESSION NUMBER: 1966:43634 CAPLUS
DOCUMENT NUMBER: 64:43634
ORIGINAL REFERENCE NO.: 64:8112e-h,8113a-h,8114a-h,8115a-c
TITLE: Chemistry of furosemide. I. Syntheses of 5-sulfamoylanthranilic acid derivatives
AUTHOR(S): Sturm, Karl; Siedel, Walter; Weyer, Rudi; Ruschig, Heinrich
CORPORATE SOURCE: Farbwerke Hoechst A.-G., Frankfurt/M., Germany
SOURCE: Chem. Ber. (1966), 99(1), 328-44
DOCUMENT TYPE: Journal
LANGUAGE: German

IT **4793-50-4**, Anthranilic acid, N-acetyl-5-(acetylsulfamoyl)-4-chloro-N-furfuryl-
(prepn. of)
RN 4793-50-4 CAPLUS
CN Anthranilic acid, N-acetyl-5-(acetylsulfamoyl)-4-chloro-N-furfuryl- (7CI, 8CI) (CA INDEX NAME)

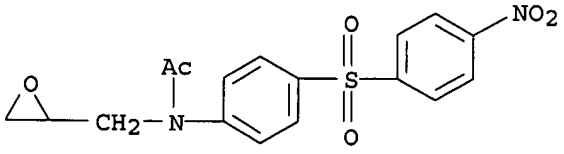


L6 ANSWER 344 OF 348 CAPLUS COPYRIGHT 2003 ACS
AB Unavailable
ACCESSION NUMBER: 1964:82598 CAPLUS
DOCUMENT NUMBER: 60:82598
ORIGINAL REFERENCE NO.: 60:14417e-f
TITLE: Reactions of Ivanov-like reagents prepared from N,N-disubstituted toluene-.alpha.-sulfonamides
AUTHOR(S): Kim, Hyun Koo
CORPORATE SOURCE: Univ. of Michigan, Ann Arbor
SOURCE: (1964) 89 pp. Avail.: Univ. Microfilms (Ann Arbor, Mich.), Order No. 64-840
From: Dissertation Abstr. 24, 3097-8
DOCUMENT TYPE: Dissertation
LANGUAGE: Unavailable

IT **93332-09-3**, Acetanilide, N-(2,3-epoxypropyl)-4'-[(p-nitrophenyl)sulfonyl]-
(prepn. of)
RN 93332-09-3 CAPLUS
CN Acetanilide, N-(2,3-epoxypropyl)-4'-[(p-nitrophenyl)sulfonyl]- (7CI) (CA INDEX NAME)



L6 ANSWER 345 OF 348 CAPLUS COPYRIGHT 2003 ACS
AB A mixt. of 5 g. 4-nitro-4'-aminodiphenyl sulfone and 2.6 g. allyl bromide in 75 ml. EtOH was refluxed 3 hrs., evapd., NaHCO3 soln. added to the residue, and the mixt. extd. with Et2O to give 3.5 g. 4-nitro-4'-allylaminodiphenyl sulfone (I), m. 110-15.degree.. Heating 5 g. I with 35 ml. Ac2O 2 hrs. gave 4-nitro-4'-allylacetylaminodiphenyl sulfone (II), m. 216-20.degree. (AcOH). To a cooled (-5.degree.) and vigorously agitated mixt. of 20 g. Bz2O2 and 300 ml. PhMe was added dropwise 50 ml. 10% EtONa in EtOH over 5 min., 350 ml. ice-H2O added, the mixt. kept with 2 g. II overnight, washed with NaHCO3 soln., evapd., and the residue washed with Et2O, and refrigerated overnight to give 0.7 g. 4-nitro-4'-(.beta.,.gamma.-epoxypropyl)acetylaminodiphenyl sulfone (III), m. 103-5.degree.. Catalytic redn. of 0.5 g. III with 0.3 g. C and 30 ml. 0.5% PdCl2 soln. 5 min. gave 0.2 g. 4-amino-4'-(.beta.,.gamma.-dihydroxypropyl)acetylaminodiphenyl sulfone (IV); picrolonate m. 135.degree. (decompn.) (H2O). A soln. of 0.2 g. III in 10 ml. EtOH was heated with aq. ethanolic HCl 30 min. to give 4-nitro-4'-acetonylaminodiphenyl sulfone, yellow, m. 141-2.degree. (EtOH). IV inhibits growth of Mycobacterium tuberculosis H37Rv.
ACCESSION NUMBER: 1964:82597 CAPLUS
DOCUMENT NUMBER: 60:82597
ORIGINAL REFERENCE NO.: 60:14417c-e
TITLE: Synthesis and antibacterial activity of 4-amino-4'-polyhydroxyalkylaminodiphenyl sulfone
AUTHOR(S): Maruyama, I. Kozo; Kawanabe, Koji
CORPORATE SOURCE: Meiji Coll. Pharm., Tokyo
SOURCE: Meiji Yakka Daigaku Kenkyu Kiyo (1963), 2, 69-72
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
IT **93332-09-3**, Acetanilide, N-(2,3-epoxypropyl)-4'-[(p-nitrophenyl)sulfonyl]-
(prepn. of)
RN 93332-09-3 CAPLUS
CN Acetanilide, N-(2,3-epoxypropyl)-4'-[(p-nitrophenyl)sulfonyl]- (7CI) (CA INDEX NAME)



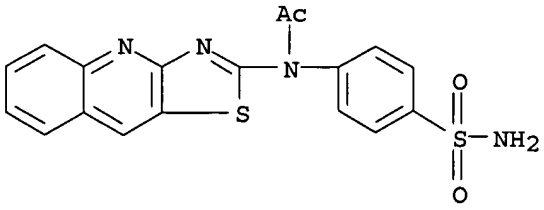
L6 ANSWER 346 OF 348 CAPLUS COPYRIGHT 2003 ACS
GI For diagram(s), see printed CA Issue.
AB cf. CA 53, 15085d; 58, 5654a. I (R = NH2) (0.2 g.) and 0.4 g. p-MeC6H4SO2Cl in 2 ml. anhyd. C5H5N gave, after several min., 0.18 g. I (R = p-MeC6H4SO2NH), m. >335.degree. (PhNO2). Similarly was prepd. I (R =

p-O2NC6H4SO2NH), m. 312-13.degree. (decompn.), (PhNH2), and I (R = p-AcNHC6H4SO2NH), m. 327-8.degree. (decompn.) (PhNO2), hydrolyzed to I (R = p-H2NC6H4SO2NH), m. 335.degree.. I (R = Cl) (0.13 g.) and 0.1 g. sulfanilamide in 3 ml. EtOH refluxed on a water bath 10 min., gave I (R = p-H2NSO2C6H4NH), m. 296-8.degree. (decompn.) (aq. EtOH); N4-acetyl deriv. (III), m. 247-8.degree. (decompn.) (EtOH). I (R = Cl) (1 g.) and 1 g. N1acetylsulfanilamide in 30 ml. EtOH refluxed on a water bath gave yellow I (R = p-H2NSO2C6H4NAc).HCl, m. 294-5.degree. (decompn.); free base, m. 316-17.degree. (decompn.) (PhNO2). III (0.43 g.) refluxed with Ac2O 60-90 min. gave the N2,N4diacetyl deriv., m. 287-8.degree. (decompn.). I (R = Cl) (0.1 g.) and ethanolamine in 1 ml. EtOH warmed 15 min. on a water bath, gave I (R = HOCH2CH2NH), 0.11 g. m. 240-2.degree. (EtOH). Also prepd. was I (R = N(CH2CH2OH)2 m. 177 9.degree. (H2O), which with SOCl2 in CHCl3 gave I (R = N(CH2CH2Cl)2].HCl, m. 216.degree. (decompn.) Similarly were prepd. the following II (R and m.p. given): p-AcNHC6H4SO2NH, 333-4.degree. (decompn.) (PhNO2); H2NC6H4SO2NH, m. 306-7.degree. (decompn.) (aq. EtOH); p-H2NSO2C6H4NH, 318-19.degree. (decompn.); N(CH2CH2OH)2, 223-4.degree. (aq. EtOH); N(CH2CH2Cl)2, (HCl salt m. 137.degree.).

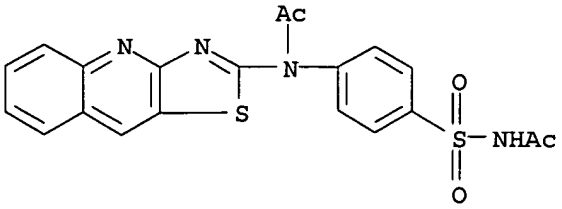
ACCESSION NUMBER: 1963:441662 CAPLUS
DOCUMENT NUMBER: 59:41662
ORIGINAL REFERENCE NO.: 59:7508d-f
TITLE: Thiazoloquinolines. VII. Novel 2-substituted derivs. of thiazolo[4,5-b]- and [5,4-b]quinolines
AUTHOR(S): Denes, V.; Ciurdaru, Gh.
SOURCE: Acad. Rep. Populate Romine, Filiala Cluj, Studii Cercetari Chim. (1962), 13(1), 89-94
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

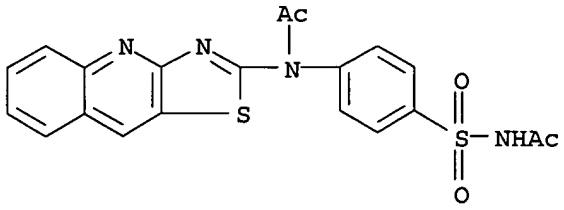
IT **98220-55-4**, Thiazolo[4,5-b]quinoline, 2-[N-(p-sulfamoylphenyl)acetamido]- **100354-24-3**, Sulfanilamide, N1,N4-diacetyl-N4-thiazolo[4,5-b]quinolin-2-yl-
(prepn. of)

RN 98220-55-4 CAPLUS
CN Thiazolo[4,5-b]quinoline, 2-[N-(p-sulfamoylphenyl)acetamido]- (7CI) (CA INDEX NAME)



RN 100354-24-3 CAPLUS
CN Sulfanilamide, N1,N4-diacetyl-N4-thiazolo[4,5-b]quinolin-2-yl- (7CI) (CA INDEX NAME)





L6 ANSWER 347 OF 348 CAPLUS COPYRIGHT 2003 ACS

AB A mixt. of 99.2 g. p-H2NC6H4SO2C6H4NH2-p (I), 155.2 g. epichlorohydrin, 28 ml. 2-methoxyethanol, and 14 ml. H2O was treated at 60.degree. for 5 days, mixed with 200 ml. MeCOEt and 121 ml. aq. 640 g./l. KOH for 2.5 hrs. at 40.degree., and H2O added. The org. layer was sepd., dild. with 200 ml. ethylene dichloride, washed with H2O, and evapd. to yield 130 g. N,N,N',N'-tetraepoxide deriv. of I of softening point (s.p.) 40.degree., epoxy value 6.44 equivs./kg. A similar resin, prepd. by digesting for 6 days and by using NaOH in place of KOH, had a s.p. of 50.degree. and epoxy value of 5.76 equivs./kg. and was used to prep. a 2-part adhesive, part (a) contg. the resin and MeOAc and part (b) contg. the resin, I, and MeOAc; the 2 parts are mixed immediately before using to bond sheets of Al, the bonded sheets having higher tensile shear strength at 260.degree. than those bonded with Bisphenol A resin, but a lower shear strength at .ltoreq.150.degree..

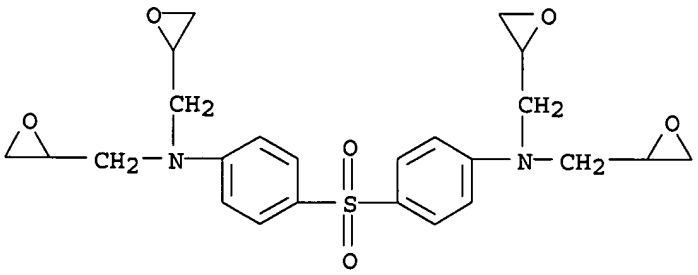
ACCESSION NUMBER: 1963:3807 CAPLUS
DOCUMENT NUMBER: 58:3807
ORIGINAL REFERENCE NO.: 58:640c-e
TITLE: Epoxy-resin adhesives from diaminodiphenyl sulfones
INVENTOR(S): Garnish, Edward W.
PATENT ASSIGNEE(S): CIBA (A.R.L.) Ltd.
SOURCE: 6 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 907844		19621010	GB	19590415

IT **95954-72-6**, Aniline, 4,4'-sulfonylbis[N,N-bis(2,3-epoxypropyl)- (adhesive manuf. from)

RN 95954-72-6 CAPLUS

CN Oxiranemethanamine, N,N'-(sulfonyldi-4,1-phenylene)bis[N-(oxiranylmethyl)- (9CI) (CA INDEX NAME)



L6 ANSWER 348 OF 348 CAPLUS COPYRIGHT 2003 ACS

AB Passing 9.1 g. dry HCl into 21 g. dihydropyran at 10.degree. and adding the 2-chlorotetrahydropyran to 33.3 g. AgCN in 125 cc. refluxing anhyd. Et2O, refluxing the mixt. 3 hrs., and evapg. the Et2O from the filtered soln. yield 38% 2-cyanotetrahydropyran (I), b22 81.5.degree., nD20 1.4425, d20 1.0128. Refluxing 6.1 g. I 5 hrs. with 5 g. NaOH in 45 cc. H2O, acidifying the soln. with 15 cc. 6N H2SO4, and extg. with Et2O give 67% tetrahydropyran-1-carboxylic acid, b24 144-7.degree., nD20 1.4661, d20 1.161. Adding 6.1 g. I in 40 cc. Et2O dropwise to 0.1 mole PhMgBr in 75 cc. Et2O, keeping the mixt. 8 hrs., pouring it onto 75 g. ice and 10 cc. concd. H2SO4, extg. the aq. layer with Et2O, and distg. the residue of the Et2O soln. yield 2-benzoyltetrahydropyran, b26 170-1.degree., nD20 1.5445, d20 1.102 (2,4-dinitrophenylhydrazone, m. 171-3.degree.). Adding 22.2 g. I in 40 cc. Et2O dropwise at 0.degree. to 7.6 g. LiAlH4 in Et2O, then adding 8 cc. H2O, 6 cc. 6N NaOH, and another 28 cc. H2O, decanting the Et2O, refluxing the ppt. 10 min. with Et2O, and distg. the residue of the Et2O exts. give 66% 2-aminomethyltetrahydropyran (II), b21 64-6.degree., nD20 1.4598, d20 1.9635. Cautiously adding 31.6 g. p-AcNHC6H4SO2Cl to 15 g. II in 20.8 g. C5H5N, heating the mixt. 45 min. at 100.degree., and pouring it into 130 cc. H2O acidified with HCl yield 30 g. N-(2-tetrahydropyranylmethyl)-4-acetamidobenzenesulfonamide (III), m. 131.5-3.5.degree., which, refluxed 1 hr. with 200 cc. 2N NaOH and the soln. neutralized with concd. HCl, gives 46% N-(2-tetrahydropyranylmethyl)-4-aminobenzenesulfonamide, flakes, m. 95-7.degree. (methiodide, 40%, m. 188-90.degree.). III is found to be inactive in vitro toward Proteus vulgaris and in vivo toward a strain of hemolytic streptococcus.

ACCESSION NUMBER: 1957:39235 CAPLUS
DOCUMENT NUMBER: 51:39235
ORIGINAL REFERENCE NO.: 51:7365d-g
TITLE: Preparation and derivatives of 2-cyanotetrahydropyran
AUTHOR(S): Nelson, Bernard A.; Hodges, Elizabeth J.; Simon, Justine I.
CORPORATE SOURCE: Wheaton Coll., Wheaton, IL
SOURCE: J. Org. Chem. (1956), 21, 798-9
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

IT **106736-58-7**, Acetanilide, 4'-sulfamoyl-N-[(tetrahydropyran-2-yl)methyl]-
(prepn. of)

RN 106736-58-7 CAPLUS

CN Acetanilide, 4'-sulfamoyl-N-[(tetrahydropyran-2-yl)methyl]- (6CI) (CA INDEX NAME)